Laboratory evaluation of two LISST-25X using river sediments

Leonardo Filippa a,*, Luana Freire b, Alfredo Trento a, Ana M. Álvarez a, Marcos Gallo b, Susana Vinzón b

a Facultad de Ingeniería y Ciencias Hídricas, Universidad Nacional del Litoral (FICH-UNL), CC 217, (3000) Santa Fe, Argentina
b Área de Engenharia Costeira e Oceanográfica, Programa de Engenharia Oceânica (PEnO)/COPPE, Universidade Federal do Rio de Janeiro (UFRJ), CT C-209 Caixa Postais 68508-21945-970 Rio de Janeiro, RJ, Brazil

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There are different methods to determine particle size, most of them are only applicable in the laboratory. In order to describe the suspended fine sediment sizes where flocs may be part of the suspension, in situ measurements are essential. This work shows the results of sediment size measurements done in the laboratory using two LISST-25X diffractometers, developed for in-situ measurements, and a Malvern Mastersizer 2000 diffractometer for laboratory use. The two LISST-25X models characterize the sample size through the Sauter Mean Diameter (SMD). Besides the SMD, the Malvern diffractometer determines the granulometric distributions of the samples. The tested samples are from different fluvial and estuarine environments (Paraná, Salado and the Amazon Rivers) and their sizes (SMD) range from 4 to 300 μm. The relationship between SMD obtained with the two LISST-25X and the Malvern diffractometers gave a determination coefficient of $R^2 = 0.98$. Compared to the Malvern instrument, used as a reference, it was observed that the LISST instruments tend to overestimate the measured SMD for diameters lower than 20 μm, and to underestimate the values of samples with larger diameters (>20 μm). Aiming to obtain other characteristic diameters of the granulometric distribution from SMD measurements obtained with LISST-25X diffractometers, correlations between SMD and $d_{50}$, $d_{32}$ and $d_{20}$ were established. The dependency of the measurement of sediment mass concentration obtained with LISST instruments, with the particle size, is also addressed.

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1. Introduction

Different methods have been developed to determine particle size (Rawle, 2010), most of them are only applicable under laboratory conditions. However, for the size characterization of suspended fine sediments with formation or presence of flocs, in situ measurements are essential. The instruments available for field particle measurements include, among others, the series of LISST (Laser In-Situ Scattering and Transmissometry) sensors: LISST-25X, LISST-100, LISST-SL and LISST-ST, Sequoia Scientific Inc. Few comparisons between in situ laser diffraction methods and other techniques are available in the literature. For instance, Eisma et al. (1996) used a Malvern field-adapted instrument deployed along with submersible video and photographic devices. It was found that Malvern underestimates sizes when compared to the other devices, due to its tendency to break the aggregates. A comparison between LISST-100 and acoustical and optical sensors made by Lynch et al. (1993) showed positive correlations between the LISST and the other instruments. Stramski (2006) also compared LISST-100 and a photographic sensor. It was found that LISST-100 is more accurate than the photographic sensor particularly for measuring small particles, because of sensor resolution limitations. The main limitation of LISST-100 mentioned in those works is the lack of accuracy when the particle size reaches the detection limits of the instrument. Agrawal and Pottsmonth (1994), Traykovski et al. (1999), Cartner et al. (2001), Agrawal et al. (2008), among others, report field and laboratory projects with more advanced equipment, LISST-100 and LISST-ST. With regard to LISST-25X, used in this project, Agrawal and Mikkelsen (2009) explained the operating principle of the equipment, and Topping et al. (2006) used this instrument to determine suspended sediment concentrations and sediment sizes in a reach of Colorado River (USA).

The operating principle of the LISST-25X instrument is based on a low angle laser light scattering proposed by Lorenz-Mie (Sequoia, 2008). According to this principle, the small particles scatter most light at big angles, whereas big particles scatter light at very small angles. The LISST-25X instrument determines the Sauter Mean Diameter of the complete sample (SMD), the Sauter Mean Diameter of the coarse fraction, $\mu SMD_{c}$, the total suspended sediment volume concentration (SSC), the coarse suspended sediment volume concentration (SSC$_{c}$), the optical transmission level (OT), and the instrument operating depth.

The SMD (also called $d_{50}$ or $D[3,2]$) is used to characterize the sizes of the suspended sediment. It is defined as the diameter of a sphere

* Corresponding author.

E-mail address: leofilippo@yahoo.com.ar (L. Filippa).
that has the same volume/surface area ratio as the particle of interest. Mathematically, it is defined according to the following equation (Pacek et al., 1998):

$$SMD = \frac{d_{\text{max}}^2}{\int_{d_{\text{min}}}^{d_{\text{max}}} d^2 p(d) \, dd}$$  

where \( d \) indicates the particle diameter, \( d_{\text{max}} \) and \( d_{\text{min}} \) indicate the maximum and minimum diameters of particle distribution, respectively, and \( p(d) \) indicates the probability density function of \( d \). The LISST-25X instrument mathematically transforms the scattering intensity into the following two variables: particle volume concentration, \( \text{SSC}^\text{vol} \), and particle area concentration, \( \text{SSC}^\text{amp} \). It then determines the sample \( \text{SMD} \) based on the following relationship between the concentrations (Agrawal and Mikkelsen, 2009):

$$\text{SMD} = 1.5 \frac{\text{SSC}^\text{vol}}{\text{SSC}^\text{amp}}.$$  

The coefficient 1.5 results from considering the surface area projected. More detailed information about the operating principle of the LISST-25X sensors as well as an introduction to the equations to calculate the output variables can be found in Agrawal and Mikkelsen (2009).

According to Sequoia Scientific Inc. (Sequoia, 2009), LISST-25X can measure: \( \text{SMD} \) between 2.50 \( \mu \)m and 500 \( \mu \)m; \( \text{SMD}_g \) between 63 \( \mu \)m and 500 \( \mu \)m; \( \text{SSC} \) and \( \text{SSC}_g \) from 0.10 to 1000 mg/L, respectively, for an optical path length of 2.50 cm; \( \text{OT} \) varies at the 0–100%, with an optimum interval of 30–98%. The optical path length is the product of the geometric length of the path that the light follows through the system, and the index of refraction of the medium through which it propagates. The \( \text{SMD} \) and \( \text{SSC} \) vary linearly according to the optical path length of the instrument. The \( \text{SMD} \) variation is directly proportional to the path length, whereas for \( \text{SSC} \) the relation is inverted. The instrument resolution is 0.025\% for \( \text{SSC} \) and \( \text{SSC}_g \), 1.0\% for \( \text{SMD} \) and \( \text{SMD}_g \), and 0.10\% for \( \text{OT} \).

Malvern Mastersizer 2000 is broadly used in laboratory work. There are a variety of applications within Malvern to measure particle sizes, including measuring emulsions, suspensions and dry powders. Some applications for sediment size determinations can be found in Murray and Holtum (1996), Dyer and Manning (1999), Mietta et al. (2009), Manning et al. (2009) and Kumar et al. (2010). Thus, the Malvern measurements will be considered in the following comparisons as a reference device.

The aim of this project was to compare the sediment sizes recorded by two LISST-25X instruments (here called “model 1” and “model 2”) with the sediment sizes measured by a Malvern Mastersizer 2000 instrument (Malvern Instruments Ltd.). The in situ devices are meant for floc size measurements. However, due to the uncertainties of creating a uniform suspension and sampling for floc size measurement, the comparison was based on single particle determinations. This comparison aims to highlight the main limitations of the in situ instruments, and their operational conditions regarding the size and concentration ranges.

Different samples of natural sediments collected in fluvial and estuarine environments were used. From this comparison correlations between \( \text{SMD} \) and diameters widely used in fluvial and maritime engineering, such as \( d_{50} \), were also established.

2. Methodology

The tests were carried out with a LISST-25X optical path length of 2.50 cm (model 1) in FICH/UNL (Argentina) and LISST-25X optical path length of 0.30 cm (model 2) in LDSC/COPPE/UFRJ (Brazil). Both instruments were immersed in an acrylic prismatic testing chamber (14.50 cm × 11.50 cm × 12 cm and a total volume of 1.35 L) provided by the manufacturer, and the equipment was mounted horizontally to avoid particle sedimentation on the sensor (Sequoia, 2009). Fig. 1 shows the experimental set up.

Thirty seven sediment samples with different size distribution and origin were tested: glass micro-spheres Whitehouse Scientific (E), brick dust (B) and sediment samples from the Paraná River, Argentina (PR); the Salado River, Argentina (S), and the Amazon River, Brazil (AM). Sodium hexametaphosphate (13.50 mL, 4%) was added as a dispersant in order to avoid flocculation. The granulometric composition and the \( \text{SMD} \) of the samples were previously determined by Malvern from the average of three consecutive measurements. The source, the sizes of the samples and the granulometric classification

![Fig. 1. Experiment set up using LISST-25X (testing chamber and magnetic stirrer).](image-url)
Note: The samples labeled with the letter F were previously sieved using an ASTM 230 sieve.

During measurements, the water instruments were diluted in distilled water continuously using a magnetic stirrer (see Fig. 1). As the coarse particle concentration increased, the mixture was also manually stirred. The prevalent fractions (see Table 1): Group 1 (sample #1 to #8), with sand; Group 2 (sample #9 to #21), composed of medium and coarse silt particles; and Group 3 (sample #22 to #37), samples with a higher content of sand. Fig. 3 shows granulometric cumulative distribution for a typical sample of each group, AMP5H3G1 (Group 1), PRF (Group 2) and S37G (Group 3).

The samples whose measurements were made using LISST instruments were diluted in distilled water filling the testing chamber. During measurements, the water–sediment mixture was stirred continuously using a magnetic stirrer (see Fig. 1). As the coarse particle concentration increased, the mixture was also manually stirred. The sediment samples with high content of coarse material (E1, S19F, S25b, B41 and B58, were tested with both LISST equipments (models 1 and 2).

Table 1 summarizes the major results of the tested samples. It shows the corresponding mean sizes (SMD), variation coefficient (CV) and OT values were determined at fixed intervals.

Model 1 was set to make a total of 40 measurements per sample at a sampling rate of 5 s, and model 2 to make a total of 20 measurements per sample at a sampling rate of 2 s. For each sediment sample, the mean values and the SMD variation coefficient (CV) were determined (CV is defined as the ratio between the standard deviation of the SMD measurement and the SMD mean value). Four samples, E1, E2, B41 and B58, were tested with both LISST equipments (models 1 and 2).

3. Results

3.1. Comparison between SMD obtained with LISST and Malvern instruments
Fig. 4 compares the SMD measured using the two LISST-25X sensors to the SMD measured with the Malvern diffractometer. The SMD ranged from 4 to 300 μm, approximately. The correlation coefficient was $R^2 = 0.98$. The PR sample was excluded from this correlation due to its high sand content (Fig. 2).

From the comparison shown in Fig. 4, it is possible to note that for smaller sizes (SMD < 20 μm) the diameters measured with LISST tend to be larger than those measured with Malvern. For instance, this was the case with B49, B41, S25bf and S28bf samples (Groups 1 and 2) because they had a high percentage of fine material, 99, 99, 85 and 82%, respectively. The maximum relative difference observed between the measured SMD was 80% (5.9 μm, for the S25bf sample). For larger sizes, the SMD measured with LISST showed lower results than those measured with Malvern. Belonging to this group (Group 3) are...
the S36, S28a, S28b, S37G, SP1 and SP2 samples with high percentages of sand, 48, 49, 51, 81, 74 and 85%, respectively. The maximum difference in this case was — 56% (36.7 μm, for the AM107 sample).

For the four samples tested using the two LISST-25X models (B41, B58, E2 and E1) a positive correlation was observed between the resulting SMD, with differences of 7% (0.7 μm), 1% (0.1 μm), 14% (3.8 μm) and 7% (6.2 μm), respectively.

The measurements were done within or close (samples PRF, E1 and E2) to the optical transmission level is observed for the tested samples — 90%. When these limits were exceeded the size measurement was not good and thus discarded.

The variability in the SMD measurements using LISST instruments was quantified. Fig. 5 shows the CV as a function of SMD. It is possible to observe that the CV increases as the sample SMD increases. The minimum and maximum CV were 0.02 (B28) and 0.43 (E1), respectively.

Regarding the coarse fraction analysis, given by LISST instruments through SMDg values, Fig. 6 shows the comparison between the SMDg and d90 (obtained with Malvern), indicating a good consistency in the results. However, as shown in Table 2, SMDg is calculated even without the presence of coarse particles (see for instance samples in Group 1), when SSCg equals zero, and consequently SMDg recordings must be discarded.

### 3.2. Sediment concentrations measured with LISST instruments

The expected inverse relationship between the SSC measurements and the optical transmission level is observed for the tested samples (Table 2). Fig. 7 presents the relationship between the SSC measured with LISST and the corresponding sizes, SMD, for each suspension concentration (actual SSC). For natural sediments, the volume concentrations obtained by LISST (μL/L) were multiplied by the density, 2.65 mg/μL, in order to get the mass concentration in mg/L. As SMD increased, a decreasing trend in the LISST SSC values was observed.

### 3.3. Correlations between SMD and granulometric percentiles (d50, d10 and d90)

In fluvial and maritime hydraulics studies the median diameter, d50, is mostly used as the characteristic diameter of the size distribution. Other diameters normally used are d10 and d90. Empirical
relationships between Malvern SMD and $d_{50}$, $d_{10}$, and $d_{90}$ also measured with Malvern, were established and shown in Figs. 8, 9 and 10. The observed correlations between the SMD versus $d_{50}$ and $d_{10}$ showed $R^2 = 0.79$ and 0.98, respectively. However for $d_{90}$ a weak correlation was observed, resulting in $R^2$ being lower than 0.5.

Once the agreement between SMD measurements using LISST instruments and Malvern was verified, it was possible to use the former relationships, displayed in Figs. 8, 9 and 10, in order to estimate the percentiles of the size distribution from the SMD measured with LISST.

When the samples with bi-modal distributions, 15 of 37, are removed from the correlations above, the SMD vs $d_{50}$ correlation improves, with an increase of $R^2$ from 0.79 to 0.86, SMD vs $d_{10}$ correlation does not change, and SMD vs $d_{90}$ correlation also improves with an increase of $R^2$ from 0.48 to 0.66.

4. Discussion

A high correlation was observed between LISST SMD and Malvern SMD, with a determination coefficient of $R^2 = 0.98$, in spite of the difference between the instruments and the experimental conditions. LISST 25X is a field instrument whereas Malvern is mostly used in
laboratory work, and the size range measured by LISST runs from 2 to 500 μm and by Malvern from 0.02 to 2000 μm.

The tendency of LISST 25X to underestimate SMD in the coarser samples could be related to the agitation conditions of the water inside the test chamber as a result of the sedimentation of the coarser particles, or due to the reduced sampling volume. While Malvern pumps a considerable amount of sample through the sensor, LISST sample volume depends on the established sampling time and external agitation conditions. These sampling conditions were assessed through the variability coefficient, which in fact showed larger variability associated with the coarse samples (CV<0.15). The better mixture for finer sediments in the testing chamber would, therefore, generate less variability. However, high CV values were also detected for finer samples, such as AMP5H3G1, S19F, AMP5H3G4 and S36F (see Table 2). However in these cases, low uniformity (d90/d10<10) was observed, which may be attributed to the influence of the coarse fraction on the low sampling volume.

A large influence of the particle size in the LISST SSC was observed, decreasing SSC as the SMD increases (Fig. 7), which can be explained by resulting volume concentrations from surface measurements by optical instruments.

The available experimental conditions did not allow for testing the floc measurement capabilities. Floc size measurements are expected to be correctly determined by diffractometer instruments since the

**Fig. 6.** Relationship between SMDg (measured with LISST) and d90 (measured with Malvern).

**Fig. 7.** Relationship between LISST SSC (measured with LISST model 1) and LISST SMD and actual SSC.
density of the particles will not interfere with the measurements. However, for suspended sediment concentration measurements, flocculation can be an extra factor limiting their usage.

5. Conclusions

The comparison between the SMD obtained with the LISST-25X (models 1 and 2) and the Malvern diffractometers showed that in samples bigger than 20 μm, the LISST instrument underestimated the values measured with the Malvern instrument; whereas, in samples smaller than 20 μm, the LISST instrument overestimated the values measured with the Malvern instrument. However, the relationship between the diffractometers was highly satisfactory, with a determination coefficient of $R^2 = 0.98$.

The observed results may indicate that where the mentioned sampling constraints do not appear, LISST-25X particle size measurements in natural water bodies improve. It is important to emphasize the need for following the recommended optical transmission, within the optimum levels: 30% to 98%, which can be restrictive for many natural environments.

![Fig. 8. Relationship between SMD and $d_{50}$, both measured with Malvern.](image1)

![Fig. 9. Relationship between SMD and $d_{10}$, both measured with Malvern.](image2)
Satisfactory correlations were established between SMD and other characteristic diameters of the granulometric distribution, such as with $d_{10}$, $d_{50}$ and $d_{90}$, all measured with Malvern. Therefore, it is possible to extend these correlations to the SMD obtained with LISST-25X and to use them to calculate the granulometric parameters of samples.

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References


\[ d_{50} = 17.5 \text{ SMD}^{0.57} \]

\[ R^2 = 0.48 \]

Fig. 10. Relationship between SMD and $d_{50}$, both measured with Malvern.